The two Cl^- anions and the two water molecules are related by a total of four hydrogen bonds (see Table 3) with all water H atoms acting as donors, with Cl(2) being the acceptor in three cases but Cl(1) only once (see Fig. 2). The differences of the anion–cation interactions in the chloride and perchlorate complex are obviously too small to cause a difference in the conformation of the macrocation. This result corresponds to the experiments of high-resolution NMR investigations in solution (to be published). In contrast, the NMR spectra of the diprotonated (2.2.2.) cryptate show a significant anion effect. Unfortunately no single crystals of these complexes suitable for X-ray structure analysis have been obtained up to now.

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References

Cox, B. G., Murray-Rust, J., Murray-Rust, P., van Troung, N. & Schneider, H. (1982). *J. Chem. Soc. Chem. Commun.* pp. 377–379.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Dietrich, H. & Dierks, H. (1970). *Messtechnik (Braunschweig)*, **78**, 184–186.

Hall, S. R. & Stewart, J. M. (1987). Editors. *XTAL2.2 Users Manual.* Univs. of Western Australia, Australia, and Maryland, USA.

Hamilton, W. C. (1959). *Acta Cryst.* **12**, 609–610.

Keller, E. (1980). *Chem. Unserer Zeit*, **14**, 56–60.

Moras, D. & Weiss, R. (1973). *Acta Cryst.* B**29**, 400–403.

Sheldrick, G. M. (1986). *SHELXS*86. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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Structure of Hexamethylhexahydropyrnopetrocarpan

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Abstract. 1,2,7a,10,11,14a-Hexahydro-3,3,7,7,9,9-hexamethyl-3*H*,7*H*,9*H*-[1]benzopyrano[8′,7′:4,5]furo-[3,2-*c*]pyrano[3,2-*g*][1]benzopyran, $C_{27}H_{32}O_4$, m.p. 422–423 K, $M_r = 420{\cdot}55$, monoclinic, $P2_1/c$, $a = 9{\cdot}984$ (2), $b = 17{\cdot}632$ (1), $c = 13{\cdot}223$ (1) Å, $\beta = 100{\cdot}57$ (1)°, $V = 2288{\cdot}25$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}220$, $D_m = 1{\cdot}235$ Mg m^{-3} (by flotation in $CCl_4/C_4H_8O_2$), λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}56$ mm^{-1}, $F(000) = 904$, $T = 296$ K, final R, wR 0·0401, 0·0506, respectively, for 2665 observed reflections. The benzofuro–benzopyran (*C*/*D*) ring junction is *cis* and the conformation is staggered looking from C(7) to C(7*a*). Three atoms in ring *F* occupy two positions each with s.o.f.'s 0·7 and 0·3. The dihedral angle between the aromatic rings [37·3 (2)°] is close to that reported for analogous antifungal-active natural products.

Introduction. Heterocyclic compounds with the basic benzofuro–benzopyran structure have two asymmetric centers [C(7*a*), C(14*a*)]. At the junction the conformation can be *H,H-cis* or *-trans*. From ^{1}H NMR, the coupling constant between two protons (7·3 Hz) characterizes the *H,H-cis* conformation. A *cis*-fused benzofuro–benzopyran (*C*/*D*) ring junction can have two possible conformations, corresponding to the relative orientations of the methyl group attached to C(7) and the H atom attached to C(7*a*); the most stable is the staggered one (Pachler & Underwood, 1967). A number of structurally related oxygen heterocyclic compounds have been reported to be biologically active, exhibiting antifungal and antitumor activity (Vanetten, 1976; Kojima, Fukushima, Ueno & Saiki, 1970). This activity may depend on the molecular geometry (Perrin & Cruickshank, 1969).

The basic interest in analysing the detailed crystal structure of the title compound by X-ray diffraction methods was to confirm the geometry of the benzofuro–benzopyran system when affected by two pyran rings attached to their sides (Fig. 1). The structure is compared with analogous reported structures.

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Experimental. 7-Hydroxy-2,2,-dimethylchroman was treated with 3,3-dimethylacrylic acid in the presence of $ZnCl_2$–$POCl_3$ to give chromanone which on sodium borohydride reduction followed by dehydration with acid gave the intermediate chromene. Subsequently, Li_2PdCl_4-catalyzed Heck arylation of the chromene with 8-chloromercurio-7-hydroxy-2,2-dimethylchroman in acetone at refluxing temperature yielded the title compound (Narkhede, 1990).

A colourless crystal of dimensions 0·45 × 0·32 × 0·15 mm was used for intensity data collection using an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation. The cell parameters were refined using 25 reflections in the range $10 \le \theta \le 37{\cdot}8°$. Two standard reflections (248 and $3\bar{6}\bar{5}$), measured every 1800 s, showed no significant change in intensity. A total of 3373 unique reflections were scaned in the ω–2θ mode, in the range $2 \le \theta \le 70°$, $0 \le h \le 10$, $0 \le k \le 19$, $-14 \le l \le 14$, out of which 3189 observed reflections with $|F_o| \ge 3\sigma|F_o|$ were used for structure solution, $R_{int} = 0{\cdot}0316$. Lp correction. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986) with CFOM = 0·044. Initial Fourier refinement of the structure showed relatively high isotropic temperature factors, and electron density peaks near C(10), C(20) and C(21) were modelled by assigning two positions for each atom with respective s.o.f.'s 0·7 and 0·3. All non-H atoms were refined anisotropically. H atoms, except two with s.o.f. 0·3, were located from difference Fourier maps and refined with anisotropic thermal parameters. In the final refinements, 451 parameters were refined using 2665 reflections with $|F_o| \ge 5\sigma|F_o|$. $R = 0{\cdot}0401$, $wR = 0{\cdot}0506$ $\{w = k/[\sigma^2(F) + g(F^2)]$, with $k = 1{\cdot}000$ and $g = 0{\cdot}00798\}$, $(\Delta/\sigma)_{max} = 0{\cdot}345$ and in final difference map $(\Delta\rho)_{max} = 0{\cdot}144$, $(\Delta\rho)_{min} = -0{\cdot}176$ e Å^{-3}. *SHELX*76 (Sheldrick, 1976) was used for structure refinements and a CYBER-180 computer was employed.

Discussion. The positional parameters and equivalent isotropic temperature factors for non-H atoms are listed in Table 1 and bond lengths and angles in Table 2.* Fig. 2 gives a view (Motherwell & Clegg, 1978) of the molecule.

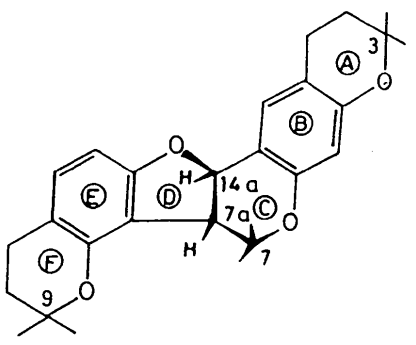

Fig. 1. Structural formula.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* U_{eq} (Å$^2 \times 10^3$) *for non-*H *atoms with e.s.d.'s in parentheses*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
C(1)	−2430 (2)	268 (1)	−427 (2)	64 (1)
C(2)	−2160 (2)	−515 (1)	−821 (1)	58 (1)
C(3)	−2276 (2)	−1134 (1)	−50 (1)	57 (1)
O(4)	−1311 (1)	−984 (1)	898 (1)	56 (1)
C(4*a*)	−1265 (2)	−260 (1)	1276 (1)	44 (1)
C(5)	−631 (2)	−180 (1)	2292 (1)	42 (1)
C(5*a*)	−515 (2)	530 (1)	2739 (1)	39 (1)
O(6)	76 (1)	561 (1)	3758 (1)	43 (1)
C(7)	698 (2)	1280 (1)	4135 (1)	38 (1)
C(7*a*)	−376 (2)	1910 (1)	3861 (1)	37 (1)
C(7*b*)	191 (2)	2706 (1)	3976 (1)	38 (1)
C(7*c*)	658 (2)	3157 (1)	4832 (1)	43 (1)
O(8)	535 (2)	2865 (1)	5768 (1)	58 (1)
C(9)	991 (2)	3322 (1)	6679 (1)	59 (1)
C(10)*	2203 (4)	3807 (2)	6579 (3)	73 (2)
C(10′)†	875 (6)	4148 (4)	6477 (5)	63 (2)
C(11)	1800 (3)	4337 (1)	5630 (2)	82 (2)
C(11*a*)	1221 (2)	3866 (1)	4703 (2)	51 (1)
C(12)	1265 (2)	4110 (1)	3713 (2)	54 (1)
C(13)	772 (2)	3689 (1)	2853 (2)	49 (1)
C(13*a*)	244 (2)	2984 (1)	3015 (1)	40 (1)
O(14)	−250 (1)	2486 (1)	2239 (1)	45 (1)
C(14*a*)	−1015 (2)	1916 (1)	2712 (1)	39 (1)
C(14*a*)	−1025 (2)	1167 (1)	2184 (1)	39 (1)
C(15)	−1642 (2)	1063 (1)	1164 (1)	46 (1)
C(15*a*)	−1769 (2)	365 (1)	687 (1)	46 (1)
C(16)	−1810 (3)	−1884 (1)	−409 (2)	82 (2)
C(17)	−3678 (3)	−1187 (2)	214 (2)	94 (2)
C(18)	1057 (2)	1155 (1)	5280 (1)	55 (1)
C(19)	1947 (2)	1406 (1)	3663 (2)	49 (1)
C(20)*	1286 (5)	2777 (2)	7578 (3)	86 (2)
C(21)†	−254 (4)	3803 (2)	6777 (3)	83 (2)
C(20′)*	227 (8)	3058 (5)	7461 (5)	86 (2)
C(21′)†	2510 (7)	3072 (4)	6901 (6)	71 (2)

* S.o.f. = 0·7.
† S.o.f. = 0·3.

The molecular skeleton may be considered to comprise two parallel strips, corresponding to three rings each (*A*, *B* and *C*; *D*, *E* and *F*) fused together at the *C*/*D* junction with an angle of 45·2 (3)°. Ring *F* is approximately in a sofa conformation (Livingstone, 1981) with the mean plane passing through the atoms C(7*c*), O(8), C(9), C(11) and C(11*a*) [maximum deviation −0·040 (3) Å] and two positions of C(10), corresponding to s.o.f.'s 0·7 and 0·3, deviate almost equally on either side of the plane by −0·706 (4) and 0·732 (6) Å, respectively. The two sites for C(10) affect the positions of the methyl groups attached to its adjacent atom [C(9)] resulting in two positions for each group with respective s.o.f.'s 0·7 and 0·3. The benzofuro–benzopyran system has a *cis* junction [C(7*a*) and C(14*a*)]. On viewing from C(7) to C(7*a*) the conformation is

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, torsion angles and least-squares-planes details have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53940 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(2)—C(1)	1·517 (3)	C(10)—C(9)	1·508 (4)
C(15*a*)—C(1)	1·510 (2)	C(10′)—C(9)	1·482 (7)
C(3)—C(2)	1·513 (3)	C(20)—C(9)	1·515 (4)
O(4)—C(3)	1·458 (2)	C(21)—C(9)	1·529 (4)
C(16)—C(3)	1·506 (3)	C(20′)—C(9)	1·468 (8)
C(17)—C(3)	1·506 (3)	C(21′)—C(9)	1·555 (7)
C(4*a*)—O(4)	1·369 (2)	C(9)—O(8)	1·452 (2)
C(5)—C(4*a*)	1·383 (2)	C(11)—C(10)	1·556 (4)
C(15*a*)—C(4*a*)	1·390 (3)	O(8)—C(7*c*)	1·366 (2)
C(5*a*)—C(5)	1·381 (2)	C(11)—C(10′)	1·612 (8)
O(6)—C(5*a*)	1·369 (2)	C(11*a*)—C(7*c*)	1·395 (3)
C(14*b*)—C(5*a*)	1·386 (2)	C(11*a*)—C(11)	1·504 (3)
C(7)—O(6)	1·459 (2)	C(12)—C(11*a*)	1·387 (3)
C(7*a*)—C(7)	1·540 (2)	C(13)—C(12)	1·373 (3)
C(18)—C(7)	1·507 (2)	C(13*a*)—C(13)	1·382 (3)
C(19)—C(7)	1·510 (3)	O(14)—C(13*a*)	1·371 (2)
C(7*b*)—C(7*a*)	1·512 (2)	C(14*a*)—O(14)	1·469 (2)
C(14*a*)—C(7*a*)	1·536 (2)	C(14*b*)—C(14*a*)	1·494 (2)
C(7*c*)—C(7*b*)	1·392 (2)	C(15)—C(14*b*)	1·388 (2)
C(13*a*)—C(7*b*)	1·371 (2)	C(15*a*)—C(15)	1·378 (3)

C(15*a*)—C(1)—C(2)	111·4 (2)	C(21′)—C(9)—C(10′)	110·7 (4)
C(3)—C(2)—C(1)	112·6 (2)	O(4)—C(3)—C(2)	109·2 (2)
C(16)—C(3)—C(2)	110·8 (2)	C(21′)—C(9)—C(20′)	113·1 (5)
C(16)—C(3)—O(4)	103·7 (2)	C(17)—C(3)—O(4)	107·9 (2)
C(17)—C(3)—C(2)	113·0 (2)	C(11)—C(10)—C(9)	108·5 (2)
C(4*a*)—O(4)—C(3)	117·1 (2)	C(17)—C(3)—C(16)	111·9 (2)
C(15*a*)—C(4*a*)—O(4)	123·4 (2)	C(5)—C(4*a*)—O(4)	115·4 (2)
C(15*a*)—C(4*a*)—C(5)	121·2 (2)	C(11)—C(10′)—C(9)	107·0 (4)
O(6)—C(5*a*)—C(5)	116·4 (1)	C(5*a*)—C(5)—C(4*a*)	119·7 (2)
C(14*b*)—C(5*a*)—O(6)	122·7 (1)	C(14*b*)—C(5*a*)—C(5)	120·9 (2)
C(7*a*)—C(7)—O(6)	108·2 (1)	C(11*a*)—C(11)—C(10)	109·3 (2)
C(18)—C(7)—O(6)	102·9 (1)	C(11*a*)—C(11)—C(10′)	105·6 (3)
C(18)—C(7)—C(7*a*)	111·7 (1)	C(11)—C(11*a*)—C(7*c*)	119·9 (2)
C(19)—C(7)—O(6)	108·6 (1)	C(12)—C(11*a*)—C(7*c*)	118·5 (2)
C(19)—C(7)—C(7*a*)	112·8 (1)	C(12)—C(11*a*)—C(11)	121·6 (3)
C(19)—C(7)—C(18)	112·0 (2)	C(13)—C(12)—C(11*a*)	123·1 (2)
C(7*b*)—C(7*a*)—C(7)	114·5 (1)	C(13*a*)—C(13)—C(12)	116·6 (2)
C(14*a*)—C(7*a*)—C(7)	112·5 (1)	C(13)—C(13*a*)—C(7*b*)	123·1 (2)
C(14*a*)—C(7*a*)—C(7*b*)	99·9 (1)	O(14)—C(13*a*)—C(7*b*)	113·2 (1)
C(7*c*)—C(7*b*)—C(7*a*)	132·5 (2)	O(14)—C(13*a*)—C(13)	123·7 (2)
C(13*a*)—C(7*b*)—C(7*a*)	108·4 (1)	C(14*a*)—O(14)—C(13*a*)	105·3 (1)
C(13*a*)—C(7*b*)—C(7*c*)	119·1 (2)	O(14)—C(14*a*)—C(7*a*)	105·8 (1)
O(8)—C(7*c*)—C(7*b*)	116·7 (2)	C(14*b*)—C(14*a*)—C(7*a*)	115·0 (1)
C(11*a*)—C(7*c*)—C(7*b*)	119·6 (2)	C(14*b*)—C(14*a*)—O(14)	111·4 (1)
C(11*a*)—C(7*c*)—O(8)	123·6 (2)	C(14*a*)—C(14*b*)—C(5*a*)	119·9 (1)
C(9)—O(8)—C(7*c*)	118·4 (1)	C(15)—C(14*b*)—C(5*a*)	117·6 (2)
C(10)—C(9)—O(8)	112·1 (2)	C(15)—C(14*b*)—C(14*a*)	122·3 (2)
C(10′)—C(9)—O(8)	113·2 (3)	C(15*a*)—C(15)—C(14*b*)	123·4 (2)
C(20)—C(9)—O(8)	106·6 (2)	C(15)—C(15*a*)—C(1)	122·4 (2)
C(20)—C(9)—C(10)	112·6 (2)	C(15)—C(15*a*)—C(4*a*)	117·3 (2)
C(4*a*)—C(15*a*)—C(1)	120·4 (2)	C(7)—O(6)—C(5*a*)	116·7 (1)
C(21)—C(9)—O(8)	104·0 (2)	C(21)—C(9)—C(10)	111·8 (3)
C(21)—C(9)—C(20)	109·2 (3)	C(20′)—C(9)—C(10′)	113·9 (4)
C(20′)—C(9)—O(8)	106·5 (3)	C(21′)—C(9)—O(8)	98·4 (3)

staggered [O(6)—C(7)—C(7*a*)—C(14*a*) 53·3 (2)°], matching the interpretation from ^{1}H NMR (Pachler & Underwood, 1967) that this is the most stable conformation, but the shapes of rings *C* and *D* are different from the above NMR interpretation. As ring *F*, the pyran ring *C* is also in a sofa conformation, with C(7) (to which two methyl groups are attached) 0·645 (2) Å out of the least-squares plane passing through the remaining five atoms [maximum deviation 0·022 (2) Å], whereas ring *A* is a half-chair, with O(4) and C(3) out [by −0·222 (1) and −0·738 (2) Å, respectively] of the least-squares plane passing through the atoms C(1), C(2), C(4*a*) and C(15*a*) [within deviation ± 0·062 (2) Å] (Demartims & Mackay, 1978). The furan ring *D* is puckered with O(14) and C(7*a*) being respectively 0·220 (1) and 0·289 (2) Å out of the plane passing through the other three atoms compared to the envelope shape in

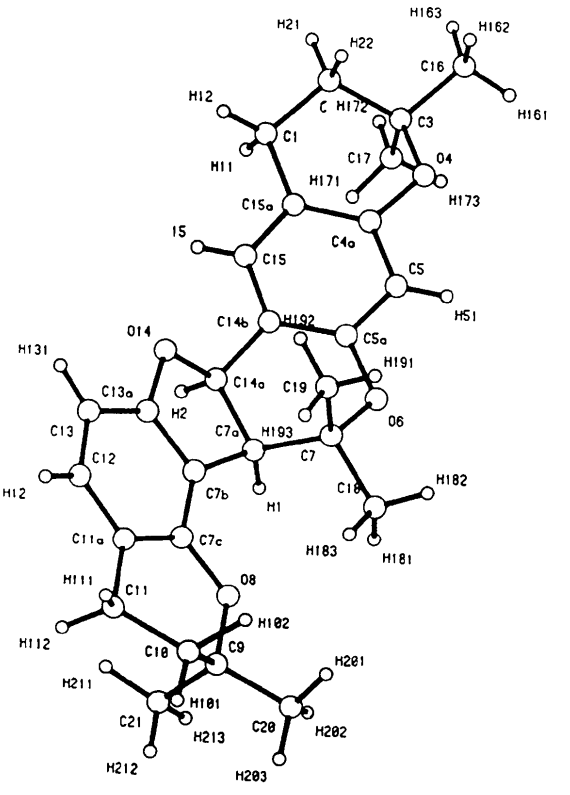

Fig. 2. General view of the molecule.

the case of 7,7-dimethyldihydropyranopetrocarpane (Das, Sinha, Narkhede & Manisekharan, 1990). Both the aromatic rings (*B* and *E*) are planar and mutually inclined at an angle of 37·3 (2)°, satisfying the suggested geometrical criteria for antifungal activity (Perrin & Cruickshank, 1969). The bond lengths and angles almost match their respective values in 7,7-dimethyldihydropyranopetrocarpane. The average C(sp^3)—C(sp^3) bond distance is 1·519 (4) Å. All intermolecular distances are longer than 3·5 Å indicating that the molecules are stabilized only by van der Waals interactions, in contrast to the intermolecular C—H···O interactions observed for 7,7-dimethyldihydropyranopetrocarpane.

References

DAS, K, SINHA, U. C., NARKHEDE, D. D. & MANISEKHARAN, T. (1990). *Acta Cryst.* C**46**, 1888–1890.

DEMARTIMS, C. & MACKAY, F. M. (1978). *Tetrahedron*, **34**, 1849–1852.

KOJIMA, R., FUKUSHIMA, S., UENO, A. & SAIKI, Y. (1970). *Chem. Pharm. Bull.* **18**, 2555–2563.

LIVINGSTONE, R. (1981). *Chroman, Alkyl- and Arylchromans.* In *Chromans and Tocopherols*, edited by G. P. ELLIS & I. M. LOCKHART. New York: John Wiley.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NARKHEDE, D. D. (1990). PhD Thesis, Indian Institute of Technology, Bombay, India.
PACHLER, K. G. R. & UNDERWOOD, W. G. E. (1967). *Tetrahedron*, **23**, 1817–1826.
PERRIN, R. P. & CRUICKSHANK, I. A. M. (1969). *Phytochemistry*, **8**, 971–978.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). In *Crystallographic Computing* 3, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.
VANETTEN, D. H. (1976). *Phytochemistry*, **15**, 655–659.

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Etude Conformationnelle du Bi(triazole-1,2,4)-3,4′

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Abstract. $C_4H_4N_6$, $M_r = 136{\cdot}11$, monoclinic, $P2_1/n$, $a = 7{\cdot}633$ (5), $b = 5{\cdot}729$ (3), $c = 12{\cdot}872$ (7) Å, $\beta = 94{\cdot}15$ (5)°, $V = 561{\cdot}5$ (2) Å^3, $Z = 4$, $D_m = 1{\cdot}65$, $D_x = 1{\cdot}61$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 11{\cdot}1$ cm^{-1}, $F(000) = 280$, room temperature, $R = 0{\cdot}036$, $wR = 0{\cdot}041$ for 1072 reflections. The two triazole rings are in envelope conformations with a dihedral angle of 8·5° between their mean planes. Molecules are held together in the crystalline state by van der Waals forces.

Introduction. Dans le cadre des recherches effectuées en séries bitriazoles (Aouial, Bernardini & Viallefont, 1977, 1978; Aouial, 1976, 1989) et afin de rendre compte des propriétés spectrales ultraviolettes du bitriazole-3,4′, une étude cristallographique de ce dernier a été entreprise dans le but de préciser la géométrie de cette molécule et de mettre en évidence d'éventuelles interactions des deaux noyaux aromatiques. Tous les cycles triazoles cités dans ce mémoire sont de type triazole-1,2,4.

On connait la structure du triazole par diffraction des rayons X soit à la température ambiante (Deuschl, 1965), soit à basse température (Goldstein, Ladell & Abowitz, 1969; Jeffrey, Ruble & Yates, 1983) ainsi que celles d'un certain nombre de bitriazoles de jonction N(4)—N(4′) (Domiano, 1977; Vreugdenhil, Haasnoot, de Graaff, Nieuwenhuis, Reefman & Reedijk, 1987). En revanche celle de bitriazole-3,4′ n'avait pas encore été résolue. Une étude par diffraction des rayons X nous a permis de définir la symétrie cristalline et la résolution de la structure du bitriazole-3,4′ à la température ambiante. Ce mémoire comporte l'exposé des résultats ainsi qu'une discussion permettant d'établir l'influence de la position relative dans l'espace des deux groupements triazoles sur la longueur de la liaison de jonction C(3)—N(4′).

Partie expérimentale. Le bitriazole-3,4′ est obtenu à partir de l'amino-3 triazole par réaction avec le *N,N*-diformylhydrazine (Wiley & Hart, 1953). La recristallisation dans un mélange à volumes égaux d'eau et de méthanol conduit à l'obtention des monocristaux transparents et de bonne qualité. La densité a été mesurée par picnomètrie dans le benzène. Le cristal utilisé pour la résolution de la structure à une forme parallélépipédique allongée suivant le direction [010] (dimensions 0,17 × 0,35 × 0,12 mm). Une étude par diffraction des rayons X à l'aide d'une chambre de Weissenberg montre que ce composé cristallise dans un système monoclinique avec les conditions de diffractions suivantes: $h0l$ $h + l = 2n$, $h00$ $h = 2n$, $00l$ $l = 2n$, $0k0$ $k = 2n$, ce qui conduit sans ambiguité au groupe d'espace $P2_1/n$. Les paramètres de la maille ont été affinés à l'aide de 25 réflexions indépendantes ($6 \le 2\theta \le 29°$) mesurées

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